N74-10292

PRECEDING PAGE BLANK NOT FILMED

Paper No. 72

SPHERE FORMING IN ZERO GRAVITY*

W. Aubin, D. Larson, Jr., G. Busch and G. Geschwind,
Research Department, Grumman Aerospace Corp.,
Bethpage, N.Y.

ABSTRACT

Melting and solidification of melted spheres in zero-gravity were studied in the Marshall Space Flight Center's 300 foot drop tower. An apparatus to melt and solidify metals in the three seconds of zero-gravity time available is described.

INTRODUCTION

The exploitation of the space environment for materials processing is very attractive. This environment, unobtainable in laboratories on earth could allow development of new materials with unique properties and improve or reduce the cost of existing materials. The most unique feature of the space environment - zero gravity - has only been considered as a potential experimental variable by scientists and engineers for a few years (Ref. 1-3). Zero gravity is difficult to realize short of suborbital or orbital flights using rockets. To provide the scientific community with the opportunity to perform experiments in zero-gravity at low cost and with a rapid turn-around time, NASA/MSFC has made available its 300 foot drop tower as a testing facility. Using this equipment it is possible to get about three seconds of very low gravity ($\sim 10^{-5}$g) test time. In order to make full use of this facility to study the effect of zero gravity on the melting and solidification of metals, it is necessary to provide an experiment in which sufficient quantities of metal can be melted and solidified in the short time available. Thus analysis can be made on the solidified material. This paper describes such an apparatus and the results

*This work was partially supported by NASA under Contract NAS 8-28604

of both laboratory and drop tower tests.

PROCESS DESCRIPTION

A survey of the literature indicated that there is a process by which metal wires can be rapidly melted and as a result of surface tension forces will form and solidify as nearly perfect spheres. With sufficiently rapid cooling, the entire process can be accomplished in less than three seconds. In the experiment a low voltage, very high current is rapidly switched across a metal wire strung between two electrodes. If the rate of power input is sufficiently high, the solid wire will melt and form a liquid column, which is unstable. While still liquid, it will decay into a chain of non-connected liquid spheres as shown in Fig. 1. This sequence is from high speed movies taken of the process in the laboratory.

While the mechanism of sphere formation is not completely understood (Ref.4 ,5,6), the process has been termed "formation of unduloids". Most simply, the wires act in the same manner as does a fuse when the line it is protecting is overloaded.

Since, at present, almost all zero gravity experiments are time limited, it is instructive to consider what is the limiting size of sphere that will form and solidify during this experiment. Assuming the process to be the decay of a liquid column to a sphere, then the decay time for this process has been given by (Ref. 7, 8):

$$\tau = \left(\frac{\rho}{\sigma}\right)^{\frac{1}{2}} R^{3/2}$$

where

ρ = density (Kg/m^3)
σ = surface tension (n/m)
$2R$ = final sphere diameter (m).

A survey of the literature indicates that ρ/σ has a value of about 0.06 for many metals at their melting points. Using this value, we have plotted the time to form spheres of various sizes and this is shown in Fig. 2. The arrows indicate the limits on the sizes of spheres that could be formed in three seconds in the drop tower (5.1 cm) and in 12 seconds in a KC-135 flight ($\sim$ 13 cm) if sufficient power is available.

It is next of interest to consider how large a sphere will solidify in the time available if we assume it is molten at the instant of achieving zero-g. This has been done for two cases (Ref. 9), both of which assume the sphere is a pure metal at its melting point. In the first case, all the heat of fusion is rejected by radiation and evaporation is neglected. The second case is for a very volatile element and assumes radiation heat transfer is small. Figure 3 presents some of the results for different metals. Consideration of Figs. 2 and 3 shows that in the time available in the drop tower, we can form very large molten spheres but they will not be completely solidified before the restoration of gravity. If large spheres are formed, then only the outer shell will have a chance to solidify before impact.

APPARATUS

Laboratory Experiments - The initial experiments in the laboratory were conducted using a bell jar vacuum system with the wire suspended from copper bus bars about one foot above the base plate. The free fall time following melting is therefore .25 seconds. AC power was supplied to the wire through a variac and a stepdown transformer; the power was switched on manually.

These experiments were carried out on several materials which were available in the laboratory. These are tabulated below with the wire diameter in centimeters. With sufficient power, it was possible to form spheres as shown in Fig. 4 from all these materials.

Metals	Alloys	Coated Wires	Other
Ag (0.025)	Nichrome (0.051)	Ni on Cu (0.015)	NiTi (0.160)
Ni (0.192)	Chromel (0.038)		
W (0.064)	Constatan (0.051)		
	SAE 1080 (0.102)		

The wide range of materials which were successfully formed into spheres indicates that the formation of the unduloids and the resultant spheres is related to the process of power input and not the materials. Since even the highest melting metal, tungsten, formed spheres, it is expected that the wire melting experiment can be used to form spheres from any metallic conductor as long as it can be fabricated into wire or rod and sufficient power can be rapidly applied.

When performing the experiment in the laboratory, using AC heating, it was observed that a threshold voltage (or power) exists below which no spheres are formed. Figure 5 shows an example of this for chromel wire. As the power (or primary voltage) increases, the number of spheres formed rises rapidly. Since the sphere diameter remains relatively constant this result indicates that more of the length of the wire forms spheres. As expected, increasing the wire diameter increased this threshold voltage, however, the shape of the curve remains the same.

The results shown in Fig. 5 are for a vacuum of 10^{-4} torr. If the pressure in the chamber is increased using inert gas, the threshold voltage also increases, but the number of spheres formed increases more rapidly with voltage at the higher pressure.

Drop Tower Experiments - The experimental apparatus designed for the Drop Tower is also compatable with Aerobee rockets for longer zero-g testing. A stainless steel can 29 cm long by 28.5 cm diameter serves as the vacuum chamber. It is connected to a portable vacuum station through a shut off valve and a quick disconnect. One end of the chamber is removable and contains the electrical feed throughs, the specimen clamps, an observation lamp, a temperature reference wire and a mirror for observing the sphere formation in two planes. This is shown in Fig. 6. Color film at 300 frames/second was used to record the experiment from a movie camera mounted at the opposite end of the chamber. The electronics necessary for the proper sequencing of events in the drop tower is mounted with the can on a frame supplied by NASA.

The evacuated experiment can, on its frame was placed in a cylindrical chamber 2.44 m in diameter and 3.05 m long in the drop tower where it is allowed to float free during the 91.5 m drop. The chamber has a hemispherical cap on the bottom which houses a bank of nickel-cadmium batteries used to provide power to the specimen, associated electronics and the telemetry transmitter during the drop. The top of the chamber is a cone and houses the pressure spheres used to thrust the entire chamber (called a drag shield) toward the ground. This thrust overcomes the air resistance of the drag shield and extends the length of zero-g test time.

Two series of drops were made one month apart using pure Ni wire as an example of a pure metal and SAE 1090 steel as an example of an important commercial alloy. The wire diameters and lengths were varied in an attempt to increase the size of the spheres.

In the first series of drops, spheres formed in four of the seven drops. When large diameter wire (.127 cm) was used at 28 volts or at 14.5 volts no spheres formed. In general, the spheres were bright and their surfaces appeared free from oxidation.

In only one drop of the second series of drops all with Ni wire were spheres successfully formed and many of the specimens appeared scaled. All of the runs made in the second series were simulated in the laboratory prior to entering the drop tower. Spheres were formed in most cases. In the section on Laboratory Experiments, the threshold power needed for sphere formation was mentioned. The laboratory tests were carried out in a vacuum of 10^{-4} torr and in an inert gas (argon) at atmospheric pressure. In both cases, there was a threshold power, but it was not markedly different.

Subsequent to the second series of drops, laboratory experiments were conducted using "Die Hard" batteries, .4 cm Ni wire five cm long, and three pressure levels: diffusion pump vacuum (1×10^{-4} torr), roughing pump vacuum ($\sim 5 \times 10^{-2}$ torr), and atmospheric pressure. At the highest and lowest pressures, spheres formed, although at atmospheric pressure they were oxidized. At the intermediate pressure, no spheres ever formed; the wire always broke into segments. The explanation for the non-formation of spheres in the second set of drops may have resulted from the outgassing or leaking of the chamber up to a critical pressure level between valve-off and dropping. This is currently under investigation.

TEMPERATURE MEASUREMENT

The temperature-time history of a sample in a melting and solidification experiment is of primary importance. In this experiment, temperature measurement is difficult because of the rapidity of heating and cooling and because of the movement of the sphere following its formation. To allow the specimen and record its temperature-time history, a photographic technique using a high speed movie camera and color film was developed (Ref. 10).

With color film it is impossible to make absolute temperature measurements without a standard. The use of a standard to produce reference temperatures in the apparatus allows for the necessary corrections on moving from the bench test calibration to the drop tower chamber.

In this technique only a single batch of film (Kodak Ektachrome EF film, type 7242) is used both for calibration and the experiments. The calibration involves photographing tungsten wires at a series of temperatures between 1173 and 2573 K in a vacuum bell jar. The temperature of wires was measured before and after exposure with an optical pyrometer. The appropriate corrections for change in wire emissivity with temperature and for bell jar absorption were made.

The film was analyzed with a microdensitometer to measure the optical density at three wavelengths corresponding to the three emulsions of the film, red (.63 μm), green (.53 μm) and blue (.44 μm). The optical density of the wire center, for each wavelength and frame, was then calculated and plotted as a function of Planck's black body radiation function, at the appropriate temperature. These calibration curves were then utilized, in conjunction with the internal reference wire set at 1773 K in the drop tower package, to yield specimen temperature data.

Figure 7 shows a typical temperature-time history for a drop tower experiment using .051 cm diameter, 6.03 cm long Ni wire. Shown are the results of calculations for the 0.530 μm and 0.630 μm wavelengths. These curves qualitatively follow the idealized time-temperature heating and cooling curves of a pure metal. The initial rapid temperature rise in Fig. 7 is due to heating of the solid and the plateau, or thermal arrest, is a result of the time required to absorb the latent heat of fusion. At this time (.012 sec) the sample is now a liquid column and super-heating begins as the column goes unstable. Unduloids are formed and these in turn form spheres. At this point, the circuit is opened and cooling ensues. Cooling progresses until the melting point is reached and then there is a thermal arrest due to the release of the latent heat of fusion.

METALLOGRAPHIC INVESTIGATION

In general, it was found that the degree of sphericity of the drop tower samples was considerably greater than that of the laboratory samples. This reflects the increased solidification time in the drop tower before contact is made with the chamber walls. Consistent with this increased time, the samples were better able to withstand the wall contact. As the thermal contact with the wall was poorer due to enhanced sphericity, the samples likely had a slower solid state cooling rate.

The surface morphologies of those samples that had the highest degree of sphericity invariably exhibited an equiaxed surface grain morphology. These grains were not always representative of the interior grain structure. That is, they were not always carried through into the interior, and sometimes the interior grain structure could be seen in addition to the equiaxed surface morphology. This is shown in Fig. 8a and is reproduced schematically in Fig. 8b. Although the internal grains are likely to be related to specific surface grains which were favorably oriented for rapid growth from both the melt and in the solid state, their relationship to these "parent" grains is not obvious. This phenomenon may be a combination of a surface artifact and a solid state grain growth phenomenon.

Classical dendritic growth was also noted on a limited number of specimens, with the dendrite arms mutually perpendicular and corresponding to $\langle 100 \rangle$ directions in the face centered cubic lattice.

The one unique microstructure originating from the zero-gravity experiments appears to originate from those samples which have a solidified shell prior to impact with the chamber wall. In this case, if the shell has sufficient strength to withstand the impact, the specimens will remain spherical or only be partially flattened and the solidification pattern will not emanate from the region of the flat. Figure 9 shows an example of this structure. The specimen has a large volume of internal porosity as predicted by solidification theories. The uniform distribution of void volume probably results from the simultaneous nucleation of a large number of grains throughout the interior of the sphere. These rapidly grow in place and the voids are a result of the volume difference between the liquid and solid states.

SUMMARY AND CONCLUSIONS

Direct resistance melting of wire specimens has proven to be a very effective way of preparing molten metal specimens in any limited time zero gravity facility. In the first series of experiments, both nickel and steel specimens were successfully melted and solidified during the three seconds of free fall. Although spheres were formed, their size was far smaller than predicted analytically. Larger spheres may result from better control of the heating circuit prior to drop or by increasing the rate at which energy is supplied to the specimens.

The effect of chamber pressure on sphere formation is complicated and may be related to the change from radiation heat transfer at low pressures to convection at some intermediate pressure. A good vacuum (10^{-4} torr) will insure sphere formation.

A film technique was developed for measuring the temperature of the wire during heating and the spheres during cooling. The temperature time history of the metal was shown to be similar to that observed in more conventional experiments.

The observed microstructures showed many of the features that would be expected when the solidification rate varies. A unique duplex structure, in which a thin shell of material solidifies first, followed by what appears to be simultaneously nucleation of the rest of the volume leaving a uniform distribution of small voids was observed.

ACKNOWLEDGEMENTS

The authors would like to thank William Poit and Cal Lottermoser of Grumman for their help in conducting the laboratory and drop tower experiments, and Lee Ramsey of NASA/MSFC for his expediting of the drop tower testing. In particular we would like to thank Mr. L. H. Berge our COR for his continued encouragement of this program.

REFERENCES

1. H.F. Wuenscher, "Unique Manufacturing Processes in Space Environment", NASA/MSFC ME-70-1, (4/70).
2. "Space Processing and Manufacturing", NASA/MSFC ME-69-1 (10/69).
3. "Manufacturing Technology Unique to Zero Gravity Environment", NASA/MSFC ME-68-1, (11/68).
4. W. Kleen, Ann. Phys., 11, 579, (1931).
5. W. G. Chace, Phy. Fluids, 2, 230, (1959).
6. J. Nasitowski in Exploding Wires, Vol. 3, p. 295, ed. by W. G. Chace and H. K. Moore, Plenum Press, N.Y. (1964).
7. H. L. Poynter, AIAA Jour., 2, 1624, (1964).
8. W. H. Steurer and D. J. Gorham, "Processes for Space Manufacturing - Definition of Criteria for Process Feasibility and Effectiveness", Final Report on our contract NAS 8-24979 (6/70).
9. J. Reichman, "Solidification of Molten Spheres in Vacuum", Grumman Research Department Memorandum, RM-544, (6/72).
10. G. Busch and T. Hilgeman, "Photographic Technique for Measuring Temperature" to be published as a Grumman Research Department Memorandum.
11. C. Li, Grumman Aerospace Corp., private communication.

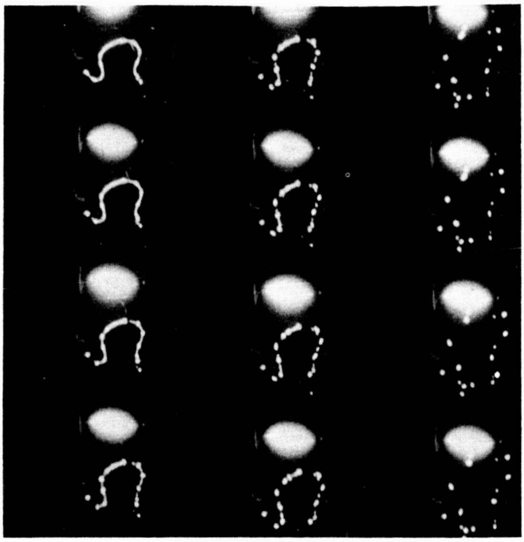


Fig. 1 Formation of Unduloids During Wire Melting

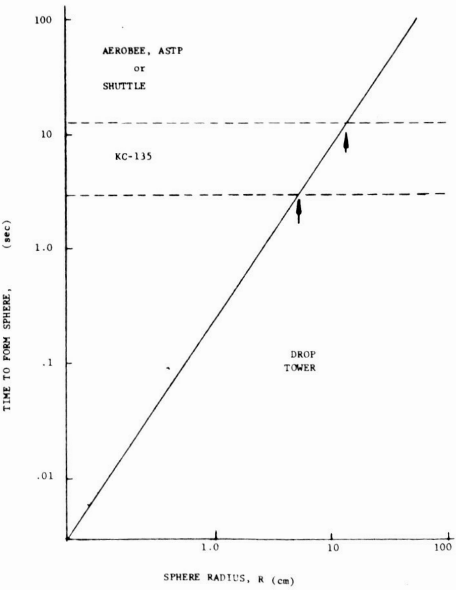


Fig. 2 Time to Form a Sphere of a Given Size

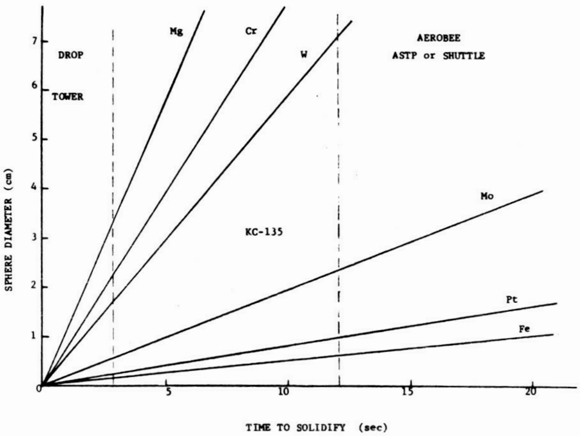


Fig. 3 Time to Solidify a Sphere of a Given Size

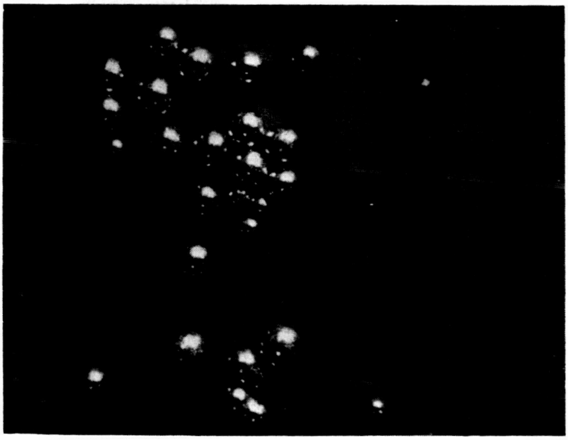


Fig. 4 Nickel Spheres Formed
in Laboratory Experiments (8X)

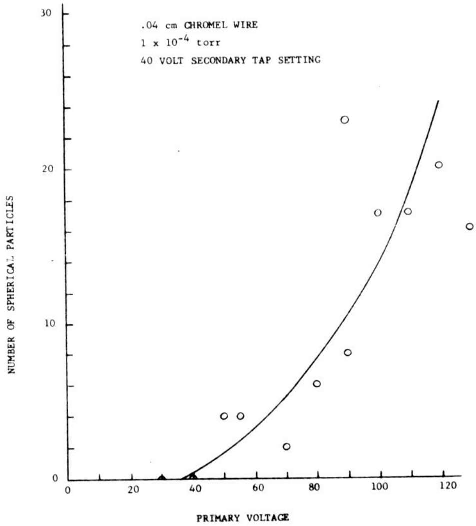


Fig. 5 Number of Spheres Formed as a Function of Power Input

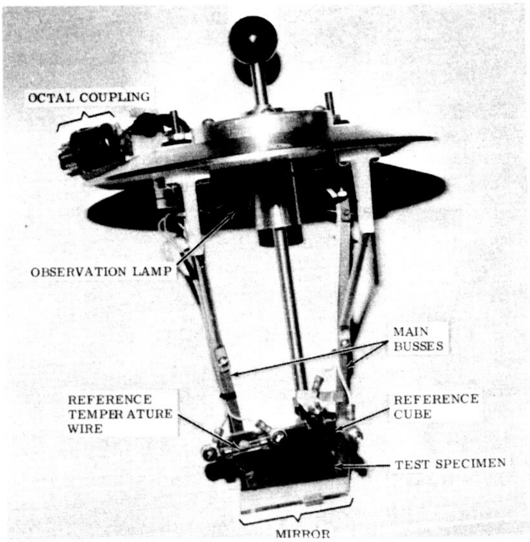


Fig. 6 Test Chamber Interior

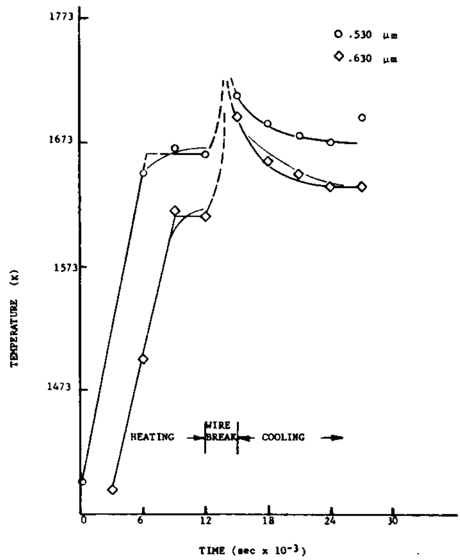
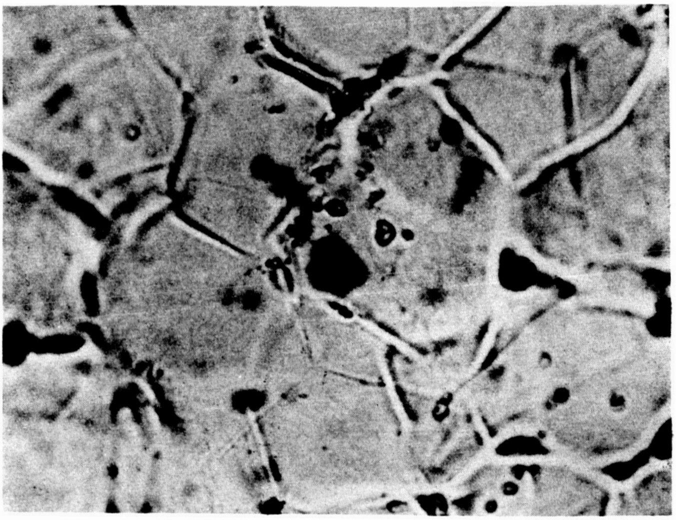
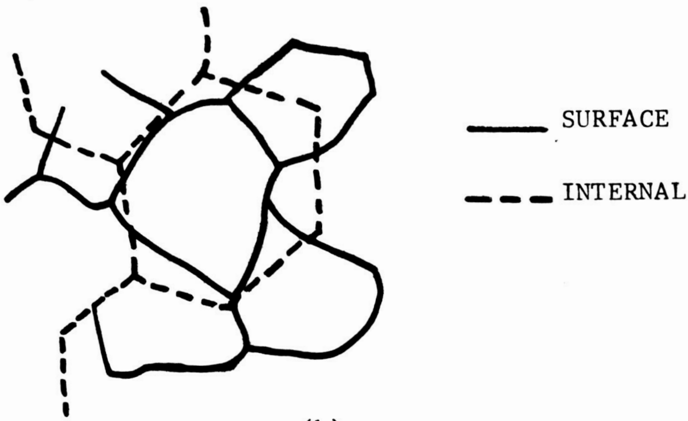


Fig. 7 Thermal History of Nickel Wire Melted in the Drop Tower



(a)



(b)

Fig. 8 Equiaxed Surface Grains and Interior Grain Structure. (a) Optical Micrograph Slightly Defocussed to Show Both Structures (375X) (b) Schematic

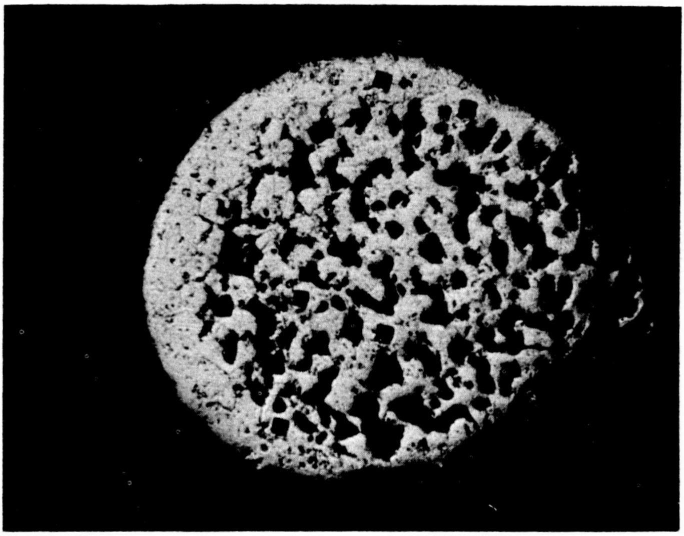


Fig.9 Internal Voids in Drop Tower Nickel Specimen (100X)

522-137 O - 73 - 59